Flow Properties of Spray-Dried Encapsulated Butteroil

R.P. KONSTANCE, C.I. ONWULATA, and V.H. HOLSINGER

ABSTRACT

The interdependence of physical properties of spray-dried butteroil encapsulated in sucrose, lactose or all-purpose flour were evaluated and compared to those of common powders such as spray-dried nonfat dry milk, whole milk powder, and sodium chloride. Powders were evaluated in terms of flow (mass flow rate and angle of repose), bulk (density) and mechanical properties (compressibility and stress relaxation). The powders were classified into three groups by mechanical sieving based on size as: ''small particles'' (<210 μm), ''particles'' (+210–420 μm) and ''encapsulated particles'' (+210–500 μm). The flow and mechanical behavior of encapsulated powders were different ($P < 0.05$) from the other powders. Encapsulated powders were less flowable ($P < 0.05$) but the addition of 2% anti-caking/flow agent enhanced flow characteristics.

Key Words: powder, flow, encapsulation, butteroil

INTRODUCTION

ENCAPSULATION is defined as the entrapment of an ingredient (solid, liquid or gas) in a continuous film or coating to provide protection to the substrate from temperature effects, moisture and pH and to prevent/retard interaction with other ingredients (Janovsky, 1993; Jackson and Lee, 1991). Encapsulation has found wide application in the food industry and is expected to show rapid growth. Encapsulation can enable the creation of products from otherwise unsuitable components. Encapsulated powders are compound matrices with ''soft'' cores that often exhibit low flow rates. Processing and handling difficulties of spray-dried butter powders containing 80% milkfat have prevented widespread use (Hansen, 1963; Prasad and Gupta, 1979; Patel et al., 1987). The use of encapsulants for production of spray-dried butter powder could enhance the handling characteristics and stability of the dry powders and provide protection from oxidative deterioration during storage (Imagi et al., 1992). These powders could then be incorporated as ingredients in other food mixtures. Their practical utility as product components rests heavily on establishing and, if required, improving their bulk and flow properties.

Bulk properties of powders are related to their physical properties and are largely influenced by powder particle size, chemical properties, and ''bridging potential.'' The ''bridging potential'' or ''stickiness'' is related to factors such as powder moisture, fat content and shape of particles (Peleg, 1983). Our objectives were to evaluate the flow properties and bulk properties of spray-dried powders containing butteroil that had been encapsulated with lactose, sucrose and all purpose flour, and to compare them to other food powders, such as lactose, sucrose, all purpose flour, salt, skim milk and whole milk.

MATERIALS & METHODS

SALT POWDERS AND GRANULATES (Norton®) were purchased locally. Other materials were obtained from suppliers as follows: sucrose (Domino Sugar Corp, NY, NY), lactose (Swiss Valley Farms, Co. Davenport, IA), all purpose flour (ADM Milling Co., Kansas City, MO), skim milk powder (Maryland & Virginia Milk Producers Association, Inc. Laurel, MD), and whole milk powder (Armour Food Ingredients, Springfield, KY). The anti-caking flow agent used was Sylox (W.R. Grace, Baltimore, MD). Butteroil was obtained from a commercial manufacturer (Land-O'-Lakes, Inc., Arden Hills, MN). Encapsulating agents were sucrose, lactose and all purpose flour. Spray-dried powders containing 40% butteroil (1.5 to 1 ratio of wall to core material) were prepared in our pilot plant as previously described (Onwulata et al., 1994a). Powder grading was done by mechanical sieving into groups. ''Small particles'' were defined as non-encapsulated materials that passed through a 210 μm sieve and ''particles,'' as those that passed through a 420 μm but not a 210 μm mesh sieve. The ''encapsulated powders'' passed through a 500 μm but not a 210 μm mesh sieve. The anti-caking agent was added at 2% to particles and encapsulated powders.

Moisture

The moisture content was determined by drying the powders in a vacuum oven at 102°C for 4 hr (AOAC, 1984) (Table 1). Moisture content was determined before and after measurement of flow properties; there was no increase ($P < 0.05$) in moisture content during or as a result of measurement of properties.

Flow properties

Powder was allowed to flow through a conical funnel at an orifice diameter of sufficient size to just permit flow. The angle of repose (θ), which determines the relative flowability of a given powder, was calculated from the base angle formed by the heap of powder

$$E(\theta) = \mathrm{Tan}^{-1} * h/r; \text{ (Sjollema, 1963)} \quad (1)$$

where h = height of powder heap (cm) and r = radius of powder heap (cm).

The mass flow of the powder (g/sec) was measured by permitting 80 g to flow through funnels of outlet diameter 1.27–2.54 cm with gentle shaking (FMC/Synthron, Homer, PA) at 40 rpm for those powders that would not flow without mechanical agitation (M-Series).

Bulk properties

The powders were carefully poured into a sample cell and the loose density (ρ_l) was determined from the weight and known volume of the cell. The sample cell, as described by Moreyra and Peleg, (1980), was 30 mm high and had a 45 mm diameter. The sample cell was mounted on the base plate of a model 4200 INSTRON Universal Testing Machine (Instron, Canton, MA). The powders were compressed at a crosshead speed of 10 mm/min using a 50 kg load cell to a preselected force of ≈ 40 KG. Irrecoverable work, or energy absorbed by the powder, was calculated from the force deformation curve using Eq. (2):

$$(A_1 - A_2)/A_1; \text{ (Moreyra and Peleg, 1980)} \quad (2)$$

where A_1 = area enclosed by compression phase and A_2 = area enclosed by decompression phase.

Powder compressibility was determined by evaluating the slope of the relationship between bulk density and the corresponding compressive stress ($1 < \log \sigma < 4$) (Fig. 1) using

$$\rho_D = a + b \log \sigma; \text{ (Sone, 1972)} \quad (3)$$

where ρ_D = bulk density (g/cm³) at corresponding σ; σ = compressive stress (g/cm²); and a,b = empirical constants with ''b'' representing compressibility.

All samples were evaluated and reported as averages of four samples. Average Coefficient of Variation (CV) for compressibility was 3.02%.

Stress relaxation of powders, which can be considered as an index of the solidity of a compressed sample, was evaluated using the INSTRON by compressing a given powder to the same strain used for its compressibility analysis (≈ 40 kg force) and measuring the stress relaxation over

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Table 1—Moisture contents of particles[e]

	Moisture (%, Wet basis)				
	S	L	F	E	FE
Salt (N)	0.05[b]	0.04[b]	0.15[a]	—	—
Sucrose (S)	0.26[c]	0.11[d]	0.80[a]	0.33[b]	0.32[b]
Lactose (L)	5.24[a]	5.11[a]	5.33[a]	4.59[b]	3.67[b]
Flour (A)	14.50[a]	15.56[a]	15.14[a]	5.97[b]	2.77[c]
Skim Milk (K)	5.37[a]	5.27[a]	5.15[a]	—	—
Whole Milk (W)	2.98[a]	2.83[a]	2.92[a]	—	—

[a-d] Superscripts that are the same within rows indicate no significant difference (P > 0.05).
[e] S = Small particles; E = Encapsulated particles; L = Particles; FE = Encapsulated particles with anti-caking/flow agent; F = Particles with anti-caking/flow agent.

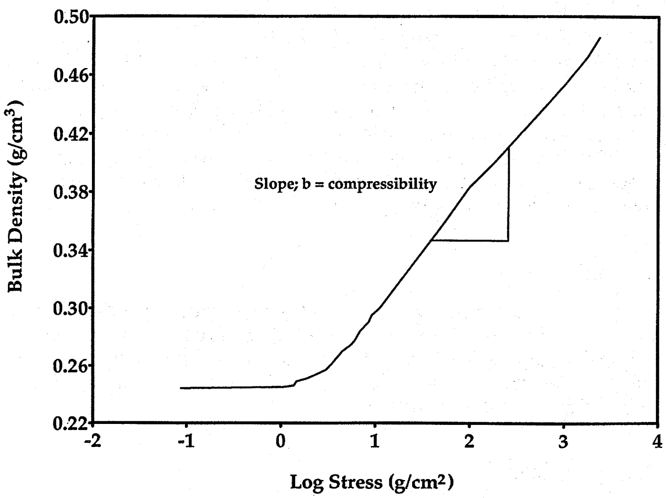

Fig. 1—Determination of powder compressibility (cm^{-1}) (b = slope of bulk density vs log stress = compressibility).

Table 2—Mechanical properties of particles[f]

	"Compressibility"[g]				
	S	L	F	E	FE
Salt (N)	0.113[a]	0.016[b]	0.037c	—	—
Sucrose (S)	0.164[a]	0.034[c]	0.032[c,d]	0.099[b]	0.027[d]
Lactose (L)	0.113[a]	0.068[c]	0.044[e]	0.079[b]	0.053[d]
Flour (A)	0.111[c]	0.118[b]	0.059[d]	0.177[a]	0.123[b]
Skim Milk (K)	0.052[b]	0.061[a]	0.027[c]	—	—
Whole Milk (W)	0.101[a]	0.096[b]	0.041[c]	—	—
	Irrecoverable Work				
	S	L	F	E	FE
Salt (N)	0.938[a]	0.444[c]	0.79[b]	—	—
Sucrose (S)	0.955[a,b]	0.847[c]	0.823[c]	0.964[a]	0.928[b]
Lactose (L)	0.899[a]	0.904[a]	0.859[b]	0.912[a]	0.907[a]
Flour (A)	0.909[a]	0.915[a]	0.870[b]	0.926[a]	0.913[a]
Skim Milk (K)	0.880[a]	0.871[a]	0.826[b]	—	—
Whole Milk (W)	0.905[a]	0.910[a]	0.766[b]	—	—
	"Relaxation"[h]				
	S	L	F	E	FE
Sucrose (S)	3.75[a]	3.46[a]	3.76[a]	2.76[b]	2.80[b]
Lactose (L)	7.58[a]	5.69[b]	5.80[b]	5.47[b]	5.14[b]
Flour (A)	2.67[b]	2.30[c]	2.10[d]	3.48[a]	2.81[b]

[a-e] Superscripts that are the same within rows indicate no significant difference (P > 0.05).
[f] S = Small particles; E = Encapsulated particles; L = Particles; FE = Encapsulated particles with anti-caking/flow agent; F = Particles with anti-caking/flow agent.
[g] "Compressibility" = b in Eq. (3) (cm^{-1}).
[h] "Relaxation" = k_2 in Eq. (4).

a 5 min period. The stress relaxation data were fit to the form of Peleg (1979) using

$$(F_o t)/(F_o - F_t) = k_1 + k_2 t \quad (4)$$

where F_o = initial force (g) and F_t = force (g) at time t and $k_1 k_2$ = constants with the slope, k_2, used as "solid" index. All samples were

Table 3—Flow properties of particles[a]

	Angle of repose				
	S	L	F	E	FE
Salt (N)	62.44	66.61	59.50	—	—
Sucrose (S)	49.23	57.72	59.59	44.73	55.82
Lactose (L)	48.33	55.35	54.20	50.38	60.24
Flour (A)	51.79	53.39	55.08	45.58	41.64
Skim Milk (K)	52.67	51.09	65.04	—	—
Whole Milk (W)	43.73	41.96	55.70	—	—

[a] S = Small particles; E = Encapsulated particles; L = Particles; FE = Encapsulated particles with anti-caking/flow agent; F = Particles with anti-caking/flow agent.

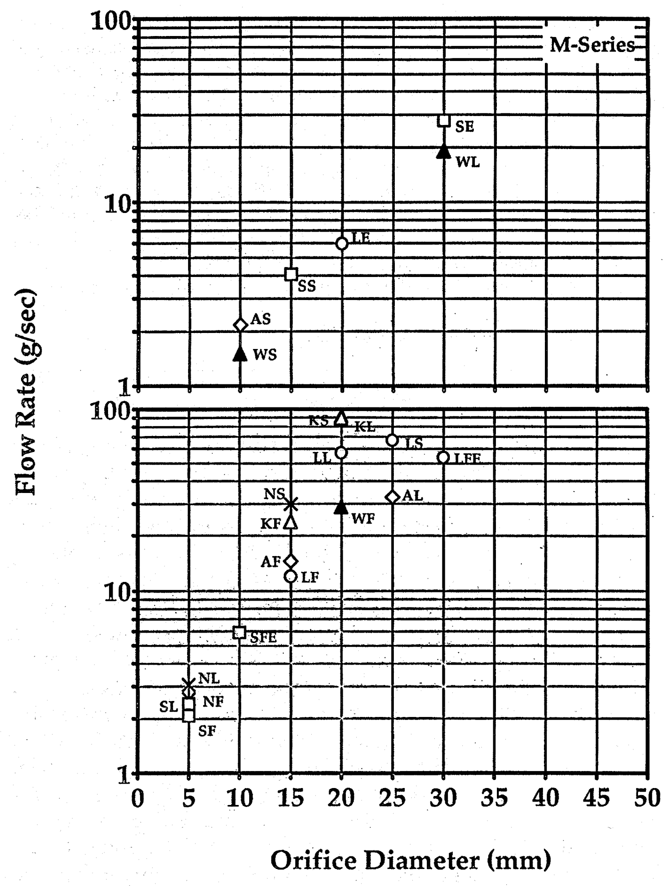

Fig. 2—Relationship of flow rate to orifice diameter. (x – salt, ○–lactose, □–sucrose, ◇–flour, △–skim milk powder, ▲–whole milk powder). (First letter of code: A = flour, L = lactose, K = skim milk, N = salt, S = sucrose, W = whole milk; second/third letter of code: S = powder, L = particulate, E = encapsulated, F = particulate with flow agent, FE = encapsulated with flow agent). M-Series are samples that were mechanically agitated.

evaluated and data reported as averages of 4 samples. Average Coefficient of Variation (CV) for solids index was 6.25%.

Tapped density (ρ_T) was determined by measuring the density of the powders after "hand-tapping" the container 100 times at ≈ 60 taps/min. Density determinations (g/cm^3) were made in triplicate.

Statistical analyses were performed using the General Linear Methods (GLM) procedure (SAS Institute, Inc., 1989). Differences were considered significant at $P < .05$. The method of principal component analysis (PCA) was used to establish relative interrelationships of the properties studied (Resurreccion, 1988). Significant correlations among variables were determined by the Proc CORR subroutine (SAS Institute, Inc., 1989).

RESULTS & DISCUSSION

Mechanical properties

Compressibility. Compressibility in many powders is a measure of internal cohesion, powder flowability and, to some

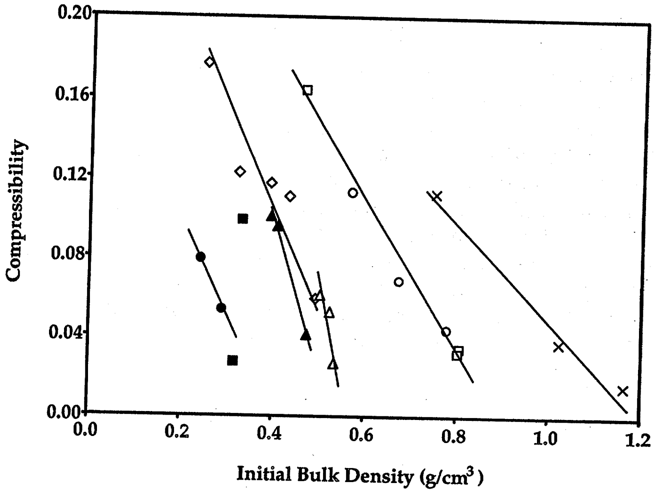

Fig. 3—Relationship of compressibility (cm^{-1}) to loose bulk density. (x–salt, ○–lactose, □–sucrose, ◇–flour, △–skim milk, ▲–whole milk, ●–lactose encapsulant, ■–sucrose encapsulant).

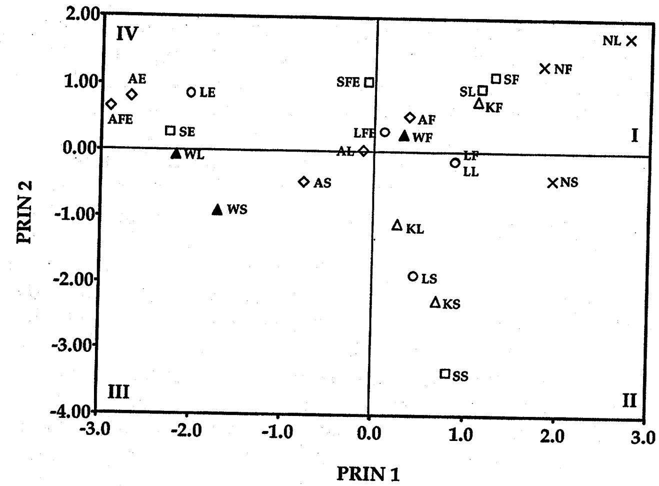

Fig. 4—Principal factor analysis of flowability. (First letter of code: A = flour, L = lactose, K = skim milk, N = salt, S = sucrose, W = whole milk; second/third letter of code: S = powder, L = particulate, E = encapsulated, F = particulate with flow agent, FE = encapsulated with flow agent.)

extent, deformability (Table 2). Carr (1976) showed that compressibility, under relatively small loads, was a sensitive index of a powder's cohesiveness and could be used to detect potential flow problems. The empirical relationship used for these determinations (Eq. 3) is valid for stresses up to 5 kg/cm² with no expectation of particle yield or breakage and the mechanism for powder bed deformation is described as particle spatial rearrangement (Peleg, 1983). For all materials studied, except salt particles (NL), there was no evidence of yield in force deformation curves. The yield points observed in the salt particle analysis indicated particle fragmentation and subsequent filling of voids. Moreyra and Peleg (1980) reported that increased powder cohesiveness resulted in decreased bulk density and an increase in compressibility. This resulted from the formation of weak and unstable open bed structures that compact/collapse easily with application of small stresses. The relationship of this compressibility to bulk density can be used to characterize a powder qualitatively and quantitatively (Peleg et al., 1973). The addition of flow agent was effective in reducing compressibility (Compare "L" to "F" and "E" to "FE") in all powders except salt and nonencapsulant sucrose. The mechanism for the behavior of these two powders was probably an interparticle filling of voids rather than spatial rearrangement and a reduction of interparticle forces. As expected, "small particle" samples ("S") were among the most compressible.

Irrecoverable work. Consistent with compressibility data, reduction of cohesiveness after addition of flow agent is indicated by the increase in recoverability of compression (Table 2). Internal friction, small amounts of particle deformation and forcing of particles into small voids are probably factors in the amount of irrecoverable work (Moreyra and Peleg, 1980). The reduction of absorbed energy is not evident for the encapsulants. Other than salt particles, where the difference is attributed to yield behavior, all other materials behaved similarly and exhibited a relatively high degree of lost work.

Stress relaxation. Stress relaxation tests are good indicators of viscoelastic behavior. Evaluation of the slope (k_2) of normalized relaxation curves (equation 3) is an index of how "solid" compacted specimens are over a short period of time. At $k_2 = 1$, liquid (viscous) behavior is expected with a resultant relaxation of stress approaching zero. Larger values of k_2 imply increased solid or elastic properties (Peleg, 1979). Data shown for relaxation (Table 2) indicated encapsulants exhibited a more viscous behavior with or without addition of flow agent. Such behavior is typical with soft powders or particles that contain fat.

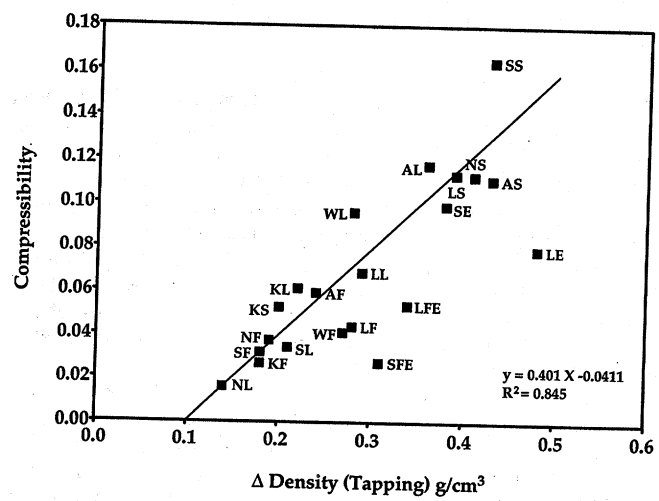

Fig. 5—Relationship between compressibility (cm^{-1}) and tapped density. (First letter of code: A = flour, L = lactose, K = skim milk, N = salt, S = sucrose, W = whole milk; second/third letter of code: S = powder, L = particulate, E = encapsulated, F = particulate with flow agent, FE = encapsulated with flow agent.)

Flow properties

Flowability of a powder is determined by both the physical properties of the powder and the geometry of the system and several experimental methods will provide indications of relative flow characteristics. The measurement of the static angle of repose, the angle formed by a heap of powder with a horizontal base, is a relatively simple method to characterize flowability (Table 3). Generally, powders exhibiting repose angles ≤ 40° are generally free flowing whereas angles > 50° indicate potential flow problems (Peleg, 1977). All samples evaluated (Table 3) had potential flow problems. The magnitude of this angle is influenced by friction and interparticle forces. The results from these measurements are, however, method dependent and Brown (1961) reported that results from different methods were not comparable.

The determination of flow rate of a powder through a horizontal orifice of varying diameters, has been used extensively in flowability determinations. Flow dynamics are affected by particle density, bulk density, particle shape and size as well as composition (White et al., 1967). A plot of log flow rate vs orifice diameter (Fig. 2) distinguishes encapsulated powders as relatively non-flowable. These powders (high fat and high moisture components) are shown in the upper portion (Fig. 3, M-Series) where mechanical agitation was required for flow. Addition of flow agent improved flow of lactose and sucrose encapsulants, as well as other food powders. Flow agent did not improve flow of all purpose flour encapsulant; no flow was observed, even when a 50-mm orifice was used. Flow properties were dependent on intrinsic properties of the powders but could not be differentiated by size or bridging potential (high fat, high moisture). The addition of flow agent increased the angle of repose for particles and encapsulants (Table 3) except for lactose (LF) and AFE (butteroil encapsulated in all purpose flour), which was not truly encapsulated (Onwulata et al., 1994b). These data show that although flow agents inherently improve flow, they do not necessarily reduce flow angle. There was an increase in flow rate (Fig. 3) and angle of repose which agreed with reported trends (Peleg and Mannheim, 1973). The effect of a flow agent on a particular powder depends on properties of the flow agent. Though Sjollema (1963) has alluded to a size-dependent influence of added flow agent, Peleg et al. (1973) reported a growth or increase in internal angle with sodium stearate. Further work is needed to investigate the observed discrepancy as well as the effect that measurement technique has on internal angle.

The relative relationship of compressibility of all powders was compared as a function of loose density (Fig. 3). Lowest bulk (loose) densities are indicative of cohesive powders with high moisture (Table 1) and/or fat content. Groupings of curves, based on material, are evident from the data. Materials in a group appear to have an intrinsic linear relation of compressibility to density. Compressibility (cohesiveness) reduction of the low moisture/low fat powders require a larger density adjustment than those with higher moisture/fat. The encapsulated powders are the most variable in density. The behavior of sucrose encapsulant supports the interparticle void filling mechanism that was postulated earlier.

Principal component analysis was used to establish relative relationships among flowabilities of powders as a function of particle size, loose density, angle of repose and flow rate. The result (Fig. 4) was a grouping of data, by quadrant, and represents an ordered, clockwise reduction in relative flow. Quadrant 1, where the highest degree of flowability occurred, is the locus for larger particle size materials and includes low moisture/low fat powders as well as those with flow agent added. Small particles and those with relatively high levels of moisture/fat appeared in quadrants 2 and 3. The encapsulated powders, which had the highest fat and/or moisture content were the least flowable and are found in quadrant 4. Encapsulated powders with added flow agent (SFE and LFE), appear to show improved flow over those without flow agent and were borderline between quadrants 4 and 1.

Compressibility was also plotted against change in density (Fig. 5) as a result of tapping. The resultant regression equation provided reasonable predictability of compressibility, normally derived from an Instron or similar equipment, based on results from a relatively simple test procedure. The regression equation included nonencapsulated powder only since the encapsulated powders appeared to behave differently.

CONCLUSIONS

SPRAY-DRIED POWDERS containing 40% butteroil showed behaviors similar to milk powders but were different than other food powders evaluated. The encapsulated powders had lower flow characteristics and, because of the fat content and the propensity for bridging, were highly cohesive. The addition of flow agent was effective in reducing cohesion and providing reasonable flow for encapsulated and nonencapsulated powders alike with exception of butteroil encapsulated in all-purpose flour. Relationships among mechanical, bulk and flow properties for nonencapsulated powders could be established, but further studies are needed to determine such relationships for encapsulated powders.

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